$[V(Et_2O)_{6-n}]^{2+}$ could possibly be used to catalyze such substitution reactions.

Conclusion

Hexacarbonylvanadium(0) reacts with 1 or more equiv of triphenylphosphine in hexane, toluene, or methylene chloride to provide high yields of (triphenylphosphine)pentacarbonylvanadium, a monomeric, paramagnetic ($\mu_{eff} = 1.87$ μ_B), and base-sensitive nonelectrolyte. This first example of a neutral monosubstituted pnicogen derivative of $V(CO)_6$ is also obtained from the oxidation of $[(C_2H_5)_4N][V(CO)_5PPh_3]$ with 1 equiv of tropylium fluoroborate.

(Triphenylphosphine)pentacarbonylvanadium(0) readily undergoes disproportionation in ethereal and more polar solvents to yield $V(CO)_5PPh_3^-$ as the only carbonyl-containing species. It has been established that the reaction of $V(CO)_6$ and triphenylphosphine in diethyl ether, originally reported by Hieber and Winter to form $[V(Et_2O)_6][V(CO)_5PPh_3]_2$, proceeds via the intermediate formation of $V(CO)_5PPh_3$. Thus, in this system, $V(CO)_6$ undergoes substitution by PPh_3 considerably faster than it is disproportionated by diethyl ether.

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Registry No. $[Et_4N][V(CO)_6]$, 56328-27-9; $V(CO)_6$, 14024-00-1; $V(CO)_5Ph_3P$, 72622-82-3; $[Na(diglyme)_2][V(CO)_6]$, 15531-13-2; $[Et_4N][V(CO)_5Ph_3P]$, 10170-61-3; $[V(Et_2O)_6][V(CO)_5PPh_3]_2$, 72622-84-5.

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Evaluation of 1,4-Dicyanobenzene as a New Internal Standard in Utilizing Solid-State Infrared Intensity Methods for the Determination of Thiocyanate Bonding Modes

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Although a wide variety of instrumental techniques[2-7] have been utilized as diagnostic tools in the determination of the bonding modes of ambidentate ligands, infrared spectroscopy[8] has proved to be the most useful, especially for thiocyanate. Despite a few inherent difficulties, the ν_{CN} bands of coordinated thiocyanate provide the most definitive criteria in this respect, in terms of their integrated absorption intensities.[8] When measured in the same solvent, these intensities vary in the order M–SCN < NCS^- < M–NCS, regardless of gross complex geometry. The use of an internal standard,[9] specifically salicylic acid, has facilitated the determination of relative ν_{CN} integrated intensity ratios, otherwise known as internal standard ratios (ISR), in the solid state.

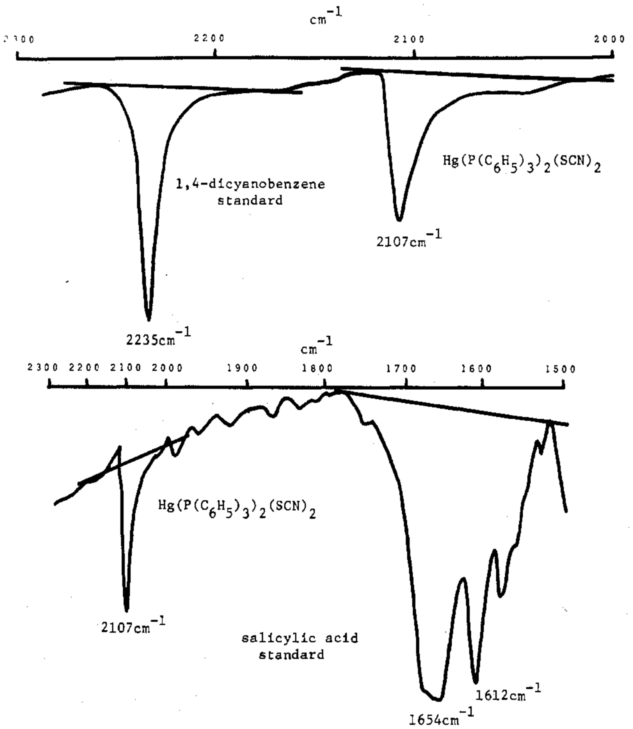

Figure 1. Representative spectra of complex and standards: upper, 1,4-dicyanobenzene standard and (dithiocyanato)bis(triphenylphosphine)mercury(II); lower, (dithiocyanato)bis(triphenylphosphine)mercury(II) and salicylic acid standard.

The previously published ISR values (see, for example, ref 8–11) have fallen into two ranges which parallel the intensity data obtained for solutions (M–SCN, <1; M–NCS, >1.5). However, the peaks in the ν_{CO} region of salicylic acid which have been used as standards (1654 cm^{-1},[8,9,11] 1612 cm^{-1} [10]) are quite irregular, making the evaluation of relative areas subject to considerable uncertainty. Furthermore, they occur in a region of the spectrum which is coincident with the absorption bands of many organic ligands. In addition, it has been shown[2,3] that the thiocyanate's bonding mode can be influenced by the surrounding environment, i.e., counterions, solvent, and physical state. Salicylic acid has the potential to significantly interact with the thiocyanate ion. Noteworthy shifts in the ν_{CN} frequencies of coordinated thiocyanates have, in fact, been observed[12,13] in the presence of salicylic acid. This, coupled with the aforementioned difficulties in evaluating relative peak areas, prompted us to search for a new internal standard. As is documented in the following discussion, we

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Table I. Thiocyanate ISR Values Obtained by Using the Nujol Mull Technique

		1,4-dicyanobenzene internal std						
				cut-and-weigh[b]				salicylic acid internal std[a]
		calcd[a]		diagonal cut[c]		straight cut[d]		
complex[j]	ν_{CN}, cm^{-1}	ISR (av)	std dev[e]	ISR (av)	std dev[e]	ISR (av)	std dev[e]	ISR
trans-$[Pd(As(C_6H_5)_3)_2(SCN)_2]$	2122	4.27	0.07	9.81	0.70	6.49	1.34	0.73[h]
trans-$[Pd(Sb(C_6H_5)_3)_2(SCN)_2]$	2113	5.65	0.51	11.0	1.3	7.32	0.49	0.81[h]
$[Pd(Et_4dien)SCN][B(C_6H_5)_4]$	2114	10.7	1.5	10.0	1.9	9.54	1.70	*f*
$[Pd(Me_4en)(SCN)_2]$	2121	35.0	5.2	15.9	1.2	26.0	2.3	2.00
$[Hg(P(C_6H_5)_3)_2(SCN)_2]$	2107	5.62	0.57	*f*	*f*	*f*	*f*	0.47
cis-$[Hg(phen)_2(SCN)_2]$	2090	6.06	0.10	*f*	*f*	*f*	*f*	0.95
salicylic acid	1654[g]	7.71	0.09	*f*	*f*	*f*	*f*	
trans-$[Pd(As(C_6H_5)_3)_2(NCS)_2]$	2092	60.7	3.1	30.3	0.4	49.4	1.8	2.6[i]
trans-$[Pd(P(C_6H_5)_3)_2(NCS)_2]$	2097	55.9	3.7	22.8	1.1	37.5	1.1	1.6[i]
$[Pd(Et_4dien)NCS][B(C_6H_5)_4]$	2092	60.5	3.8	31.3	2.0	43.2	1.0	*f*
$[Pd(bpy)(NCS)_2]$	2105	48.9	2.6	19.5	2.0	30.1	2.1	2.7[i]
trans-$[Ni(P(C_6H_5)_3)_2(NCS)_2]$	2084	30.2	3.2	*f*	*f*	*f*	*f*	3.66
trans-$[Ni(H_3CP(C_6H_5)_2)_2(NCS)_2]$	2110	20.1	2.9	*f*	*f*	*f*	*f*	1.92

[a] Calculated according to eq 1 and 2. [b] Calculated according to eq 3 and 2. [c] Base line coincident with tops of both sides of peak. [d] Base line shifted at one side, as necessary, to produce a horizontal line. [e] Minimum of three measurements. [f] Not determined. [g] ν_{CO}. [h] Reference 10. [i] Reference 11. [j] Abbreviations: Et_4dien = 1,1,7,7-tetraethyldiethylenetriamine; Me_4en = 1,1,4,4-tetramethylethylenediamine; phen = 1,10-phenanthroline; bpy = 2,2′-bipyridine.

feel that the use of 1,4-dicyanobenzene offers a number of advantages which should make it the internal standard of choice for future ISR measurements involving the thiocyanate ion.

Experimental Section

All of the complexes investigated involve thiocyanates whose bonding modes are known and have previously been reported in the literature. Infrared spectra of Nujol mulls of the complexes were measured on Perkin-Elmer 180 and 599 spectrophotometers by using a 10× abscissa expansion. In a routine analysis of this type, 30.0–150 mg of complex and a like amount of the internal standard were weighed out and combined in an alundum mortar. The mixture was ground thoroughly. One or two drops of Nujol was then added, and the mixture was ground again to ensure homogeneity. A portion of this mull was transferred to NaCl plates for the recording of the spectrum.

Infrared spectra of KBr disks of the complexes were measured on a Perkin-Elmer 180 spectrophotometer by using a 10× abscissa expansion. Approximately 5.0 mg of sample, 5.0 mg of the internal standard, and 800 mg of spectral grade KBr were thoroughly mixed for 1 min in a Wiggle-Bug before being pressed into a disk for the recording of the spectrum.

Figure 1 illustrates the peaks and measurements concerned in these experiments for both salicylic acid and 1,4-dicyanobenzene. The ISR values were determined in two different ways. In the first method, the absorption bands of both the complex (ν_{CN}) and the standard (ν_{CO} for salicylic acid, ν_{CN} for 1,4-dicyanobenzene) were assumed to be Lorentzian in shape. The integrated absorption intensity (IAI) of each band was then calculated according to

$$\mathrm{IAI} = \{\Delta\nu_{1/2}[\ln(T_{\max}/T_{\min})] \times (\text{mol wt of sample})\}/\{(\text{grams of sample}) \times (\text{no. of absorbing groups per molecule})\} \quad (1)$$

The ISR is defined as

$$\mathrm{ISR} = \mathrm{IAI}_{\text{complex}}/\mathrm{IAI}_{\text{std}} \quad (2)$$

The term $\Delta\nu_{1/2}$ in eq 1 is the peak width, in cm^{-1}, at half-height.

In the second method, the absorption bands of both the complex and the standard were cut out and weighed. The IAI for each peak was then defined according to eq 3, and the ISR values were calculated according to eq 2.

$$\mathrm{IAI} = \{(\text{wt of sample peak}) \times (\text{mol wt of sample})\}/\{(\text{grams of sample}) \times (\text{no. of absorbing groups per molecule})\} \quad (3)$$

The KBr disk spectra exhibited base lines which were relatively flat. However, as is shown in Figure 1, those of the Nujol mull spectra frequently sloped, to varying degrees. This prompted a final variation wherein, for the second method, the base lines were adjusted at one end so as to give a reasonably horizontal line, and ISR values were determined by using both the unadjusted and adjusted base lines, referred to as diagonal cut and straight cut, respectively.

Table II. Thiocyanate ISR Values Obtained by Using the KBr Disk Technique and 1,4-Dicyanobenzene as the Internal Standard

		calcd[a]		cut-and-weigh[b]	
complex[c]	ν_{CN}, cm^{-1}	ISR (av)	std dev	ISR (av)	std dev
trans-$[Pd(As(C_6H_5)_3)_2(SCN)_2]$	2121	3.6	0.0	7.5	1.6
trans-$[Pd(Sb(C_6H_5)_3)_2(SCN)_2]$	2114	5.4	1.9	9.6	4.2
$[Pd(Et_4dien)SCN][B(C_6H_5)_4]$	2115	9.4	2.0	13	2
$[Pd(Me_4en)(SCN)_2]$	2121	25	5	18	1
trans-$[Pd(As(C_6H_5)_3)_2(NCS)_2]$	2091	28	7	21	5
trans-$[Pd(P(C_6H_5)_3)_2(NCS)_2]$	2096	23	3	23	4
$[Pd(Et_4dien)NCS][B(C_6H_5)_4]$	2096	28	1	28	4
$[Pd(bpy)(NCS)_2]$	2100	22	2	17	3

[a] Calculated according to eq 1 and 2. [b] Calculated according to eq 3 and 2. [c] Abbreviations: see footnotes to Table I.

Results and Discussion

The ISR values determined by using 1,4-dicyanobenzene as an internal standard are shown in Tables I and II. The values listed in Table I originated from Nujol mull spectra, while those given in Table II were obtained by using KBr disks. Where available, ISR values utilizing salicylic acid as the internal standard have also been included for comparison purposes.

Our initial choice of 1,4-dicyanobenzene as a potentially superior internal standard was based on the following observations: (1) the ν_{CN} band of 1,4-dicyanobenzene (2235 cm^{-1}) is much sharper and more symmetrical than the ν_{CO} band(s) of salicylic acid (see Figure 1); (2) unlike the overlapping ν_{CO} bands of salicylic acid, the ν_{CN} band of 1,4-dicyanobenzene is isolated in the standard's IR spectrum; (3) the ν_{CN} band of 1,4-dicyanobenzene is found in a region of the IR spectrum which is generally devoid of other ligand absorption bands; (4) the ν_{CN} band is close (within ~250 cm^{-1}) to the ν_{CN} absorption band(s) of the thiocyanate complexes under study, permitting (in most cases) the recording of the ν_{CN} bands of both the standard and the complex without the spectrophotometer undergoing a potentially error-introducing grating change at 2000 cm^{-1} (this cannot be done by using salicylic acid); (5) 1,4-dicyanobenzene is much less chemically reactive than salicylic acid (the former is a very weak Lewis base, and its Brønsted acidity is extremely low), and, consequently, it has much less of a tendency to interact with either the metal or the thiocyanate ion.

In addition to the foregoing, an unexpected dividend was realized in practice in that the range of observed ISR values was expanded considerably, relative to that obtained with salicylic acid as the internal standard (see Table I). This tends to minimize the importance of errors in measurement, insofar as bonding mode assignments are concerned.

The data in Tables I and II clearly demonstrate, with one significant exception, the efficacy of using 1,4-dicyanobenzene as an internal standard for thiocyanate bonding mode determinations, irrespective of the calculational technique employed. The inflated ISR values exhibited by $[Pd(Me_4en)(SCN)_2]$, using both standards, are undoubtedly due to slightly nondegenerate, but unresolvable, in-phase and out-of-phase ν_{CN} stretching bands which artificially enhance the half-bandwidth. The complex has unequivocally been shown to contain S-bound thiocyanates by ^{14}N NQR measurements.[6] This illustrates the major weakness of the ISR technique, i.e., the problems encountered in dealing with overlapping, poorly resolved ν_{CN} bands. Although the ISR values to be compared should be determined by the same calculational technique, it is true, nonetheless, that, with the exception noted, M–SCN complexes generally exhibit ISR values <10, using 1,4-dicyanobenzene as the internal standard, while those of M–NCS complexes generally exceed 20. These values roughly reflect the salicylic acid ISR range limits multiplied by the ISR of salicylic acid itself (relative to 1,4-dicyanobenzene).

For optimization of the precision and accuracy of the measurements, the standard and complex ν_{CN} peak should cover as much as possible of the 80–20% transmittance range but should not exceed these limits. The cut-and-weigh method encounters more problems in dealing with samples containing small amounts of isomeric impurities, since they enhance absorption in the wings of the main peak and are thereby included in determining the weight of the peak. Unless these isomeric impurities affect $\Delta\nu_{1/2}$ and/or are a large percentage of the sample by weight, the calculation method (eq 1) is largely unaffected, since the peak is assumed to be Lorentzian in shape. The extra significant figure shown for the Nujol mull ISR values (Table I) simply reflects the larger sample size used in their preparation. The Nujol method is advantageous in that it avoids the possibility of pressure-[2] or KBr-induced[12] linkage isomerization.

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Registry No. 1,4-Dicyanobenzene, 623-26-7.

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Gas Chromatographic Separation of Facial and Meridional Tris(1,1,1-trifluoro-2,4-pentanedionato)molybdenum(III)

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Among the known oxidation states of molybdenum, the paucity of chemical information related to Mo(III) is remarkable, especially when contrasted with the rich chemistry of Cr(III). We have identified fewer than 30 isolated and thoroughly characterized complexes of molybdenum(III).[1-19] Most of these are monomeric six-coordinate high-spin (μ(eff) = 3.84) species readily described within the confines of crystal field theory. It has commonly been assumed that ligand substitution reactions of molybdenum(III) are slow, by analogy with the known chemistry of chromium(III), and under the assumption that Basolo and Pearson's argument relating crystal field splitting to the activation energy of dissociative processes may be applied to the $4d^3$ electronic configuration as readily as it has been applied to the $3d^3$ system. Indeed, Wentworth[20] dismissed the biological role of Mo(III) largely

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